भारतीय मानक

Indian Standard

IS 2697 : 2023

खाद्य उद्योग के लिए अमोनियम बाइकार्बोनेट — विशिष्टि

(दूसरा पुनरीक्षण)

Ammonium Bicarbonate for Food Industry — Specification

(Second Revision)

ICS 67.220.20

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July 2023 **Price Group 6**

Food Additives Sectional Committee, FAD 08

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

Ammonium bicarbonate is an important material for the food industry; in particular, the biscuit industry uses it in substantial quantity. This standard is intended to guide the manufacturers and the consumers of this material in producing and selecting, respectively, the material of the right quality.

This standard was first published in 1964 and thereafter first revision of standard was issued in 1976. In first revision, more stringent limits for chlorides and copper content were specified, and the methods for determination of ammonium bicarbonate, and copper were modified.

In this revision, the following major changes have been made:

a) The requirements for ammonium bicarbonate content, chlorides, sulphates, and non-volatile matter have been aligned with corresponding JECFA (1985) Monograph.

b) The requirements for heat test, and solubility have been introduced in line with JECFA (1985) Monograph, and the requirement for tarry matter has been removed from the standard.

d) The requirement for Heavy Metal (as Pb) is replaced by Lead (as Pb).

e) It also incorporates one amendment issued to this standard.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

AMMONIUM BICARBONATE FOR FOOD INDUSTRY — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for ammonium bicarbonate for food industry.

2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

IS No.	*Title*
IS 264 : 2005	Nitric acid — Specification (*third revision*)
IS 265 : 2021	Hydrochloric Acid — Specification (*fifth revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 1699 : 1995	Methods of sampling and test for food colours (*second revision*)
IS 2088 : 1983	Methods for determination of arsenic (*second revision*)
IS 2317 : 1975	Method for gravimetric determination of sulphates (*first revision*)
IS 4905 : 2015	Random sampling and randomization procedures (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white crystals or a fine white crystalline powder. Chemical name for Ammonium Bicarbonate is ammonium hydrogen carbonate.

3.2 The material shall comply with the requirements given in Table 1, when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in co1 **(4)** of the Table 1.

3.3 Heat Test

When heated, it shall volatilize without charring and the vapour shall be alkaline to moist litmus.

3.4 Solubility

The material shall be freely soluble in water, and insoluble in ethanol.

4 PACKING

The material shall be packed and supplied in polythene-lined steel drums or polythene-lined gunny bags or as agreed to between the purchaser and the supplier.

5 MARKING

5.1 Each container shall be marked legibly to give the following information:

a) Name of the material including the words 'Food Grade';
b) Source of manufacture;
c) Net quantity when packed;
d) Batch or code number;
e) Date of manufacture;
f) Instruction for storage;
g) Best before date (month and year to be given by the manufacturer); and
h) Any other requirements as specified under the *Legal Metrology (Packaged Commodities) Rules*, 2011 and *Food Safety* and *Food Safety and Standards (Packaging) Regulations,* 2018 *and Food Safety and Standards (Labelling and Display) Regulations,* 2020.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provision of *Bureau of Indian Standards Act,* 2016 and Rules and

Regulation framed thereunder and the product(s) may be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn and their criteria for conformity shall be determined in accordance with the methods prescribed in Annex B.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities that affect the results of analysis.

Table 1 Requirements for Ammonium Bicarbonate for Food Industry

(*Clause* 3.2)

Sl No. (1)	**Characteristic** (2)	**Requirement** (3)	**Method of Test, Ref to** (4)
i)	Ammonium bicarbonate, percent by mass, *Min*	99.0	**A-1**
ii)	Chlorides (as Cl), percent by mass, *Max*	0.003	**A-2**
iii)	Sulphates (as SO_4), percent by mass, *Max*	0.007	**A-3**
iv)	Iron (as Fe), percent by mass, *Max*	0.004	**A-4**
v)	Non-volatile matter, percent by mass, *Max*	0.05	**A-5**
vi)	Lead (as Pb), parts per million, *Max*	2.0	IS 1699
vii)	Copper (as Cu), parts per million, *Max*	5.0	**A-6**
viii)	Arsenic (as As), parts per million, *Max*	0.6	**A-7**

ANNEX A

(*Clause* 3.2 *and Table* 1)

ANALYSIS OF AMMONIUM BICARBONATE FOR FOOD INDUSTRY

A-1 DETERMINATION OF AMMONIUM BICARBONATE

A-1.1 Reagents

A-1.1.1 *Standard Hydrochloric Acid* — 1 N and 0.1 N

A-1.1.2 *Methyl Orange Indicator*

Dissolve 0.1 g of methyl orange in 100 ml of water.

A-1.1.3 *Thymol Blue Indicator*

Warm 0.1 g of thymol blue with 4.3 ml of 0.05 N sodium hydroxide solution and 5 ml of rectified spirit until dissolved. Dilute with 20 percent rectified spirit to 250 ml.

A-1.2 Procedure

Weigh accurately 4 g of the material and dissolve it in a 500 ml conical flask with the aid of 100 ml of freshly boiled and cooled water. Titrate the solution with standard hydrochloric acid (1 N) in the beginning and with 0.1 N standard hydrochloric acid towards the end using methyl orange indicator or thymol blue indicator. Convert the volume of hydrochloric acid (0.1 N) used into standard hydrochloric acid (1 N).

A-1.3 Calculation

Ammonium bicarbonate, percent by mass = $\frac{7.9\,VN}{M}$

where

V = volume, in ml, of standard hydrochloric acid (1 N) required for titration,

N = normality of standard hydrochloric acid, and

M = mass, in g, of the material taken for test.

A-2 DETERMINATION OF CHLORIDES

A-2.1 Reagents

A-2.1.1 *Concentrated Nitric Acid* — conforming to IS 264

A-2.1.2 *Standard Silver Nitrate Solution* — 0.1 N

A-2.1.3 *Nitrobenzene*

A-2.1.4 *Standard Ammonium Thiocyanate Solution* — 0.1 N

A-2.1.5 *Ferric Ammonium Sulphate Indicator* — approximately 5 percent

A-2.2 Procedure

Weigh accurately 20 g of the material, dissolve in water and neutralize with concentrated nitric acid and then add about 5 ml in excess. Boil the solution to expel any dissolved carbon dioxide gas, cool and add 10 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene, shake vigorously and titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate indicator.

A-2.3 Calculation

Chlorides (as Cl), percent by mass =

$$\frac{3.546\,(10\,N_1 - VN_2)}{M}$$

where

N_1 = normality of standard silver nitrate solution;

V = volume, in ml, of standard ammonium thiocyanate solution used in the titration;

N_2 = normality of standard ammonium thiocyanate solution; and

M = mass, in g, of the material taken for the test.

A-3 DETERMINATION OF SULPHATES

A-3.1 Procedure

Dissolve about 15 g of the material, accurately weighed, in about 100 ml of water. Determine the sulphates present as directed in IS 2317.

A-4 TEST FOR IRON

A-4.1 Apparatus

A-4.1.1 *Nessler Cylinders* — of 50 ml capacity

A-4.2 Reagents

A-4.2.1 *Concentrated Hydrochloric Acid* — conforming to IS 265

A-4.2.2 *Ammonium Persulphate*

A-4.2.3 *Butanolic Potassium Thiocyanate Solution*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-4.2.4 *Dilute Sulphuric Acid* — 10 percent (*v*/*v*)

A-4.2.5 *Standard Iron Solution*

Dissolve 0.702 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2$ $SO_4.6H_2O$] in 10 ml of dilute sulphuric acid, add 1 g of ammonium persulphate and heat to boiling. Cool and dilute with water to 1 000 ml. Further dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution is equivalent to 0.01 mg of iron (as Fe).

A-4.3 Procedure

A-4.3.1 Dissolve 1.000 g of the material in 25 ml of water and neutralize with concentrated hydrochloric acid. Add 30 mg of ammonium persulphate and boil to oxidize the iron. Cool and transfer completely into a Nessler cylinder. Add 15 ml of butanolic potassium thiocyanate solution and shake vigorously for 30 seconds. Carry out a control test in another Nessler cylinder using 4 ml of standard iron solution and the same quantities of other reagents. Compare the colour in the butanol layers.

A-4.3.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with material is not greater than that produced in the control test.

A-5 DETERMINATION OF NON-VOLATILE MATTER

A-5.1 Procedure

Weigh accurately about 10 g of the material in a tared porcelain or silica dish. Heat over a strong flame, cool and weigh till consecutive weighings differ by not more than ± 1 mg.

A-5.2 Calculation

$$\text{Non-volatile matter, percent by mass} = 100 \times \frac{M_1}{M_2}$$

where

M_1 = mass, in g, of the residue; and
M_2 = mass, in g, of the material taken for the test.

A-6 TEST FOR COPPER

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — of 50 ml capacity

A-6.2 Reagents

A-6.2.1 *Concentrated Hydrochloric Acid* — conforming to IS 265

A-6.2.2 *Concentrated Nitric Acid* — conforming to IS 264

A-6.2.3 *Citric Acid*

A-6.2.4 *Dilute Ammonium Hydroxide Solution* — approximately 5 N

A-6.2.5 *Sodium Diethyldithiocarbamate Solution*

Dissolve 1.0 g of sodium diethyldithio-carbamate in 1 000 ml of copper-free water. Filter and keep in an amber bottle and protect from strong light.

A-6.2.6 *Standard Copper Solution*

Dissolve 0.392 8 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) in water and make up the volume to 1 000 ml. When required for use, pipette 100 ml of the solution into a 1 000 ml flask and dilute up to the mark with water. One millilitre of this solution contains 0.01 mg of copper (as Cu).

A-6.3 Procedure

Weigh 10.000 g of the material and dissolve it in about 20 ml of water. Neutralize with concentrated hydrochloric acid and add 4 drops to 5 drops of concentrated nitric acid. Boil and cool. Add 1 g of citric acid and adjust *p*H to 9 by adding dilute ammonium hydroxide. Add 10 ml of sodium diethyldithiocarbamate solution. Dilute to the mark and mix well. In the other Nessler cylinder, carry out a control test using 5 ml of standard copper solution in place of the material.

The material shall be considered as not having exceeded the limit prescribed in Table 1, if the intensity of the colour produced with the material is not greater than that produced in the control test.

A-7 TEST FOR ARSENIC

A-7.1 Preparation of Solution

Dissolve 1.0 g of the material in minimum amount of dilute hydrochloric acid (approximately 5 N).

A-7.2 Carry out the test for arsenic with the solution prepared in **A-7.1**, as directed in IS 2088, using 0.001 5 mg of arsenic trioxide for comparison standard.

ANNEX B

(*Clause* 6)

SAMPLING OF AMMONIUM BICARBONATE FOR FOOD INDUSTRY

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.4 The samples shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-1.6 Samples shall not be taken in a place exposed to weather.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number of containers (*n*) to be chosen from a lot shall depend on the size of lot (*N*) and shall be in accordance with col (1) and (2) of Table 2.

B-2.3 These containers shall be selected at random from the lot and, in order to ensure the randomness of selection, random sampling procedures given in IS 4905 may be followed.

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **3** and shall not exceed 1 kg.

Table 2 Number of Containers to be Selected

(*Clause* B-2.2)

Sl No. (1)	Lot Size N (2)	No. of Containers to be selected n (3)
i)	Up to 50	3
ii)	51 to 200	4
iii)	201 to 400	5
iv)	401 to 650	6
v)	651 to 1 000	7

B-3.1.2 Mix thoroughly all portions of the material drawn from the same container. Out of these portions, a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stopers and labelled with all the particulars of sampling given under **B-1.5**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

B-3.2 Referee Sample

The referee sample shall consist of a composite sample (*see* **B-3.1.2**) and set of individual samples (*see* **B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of ammonium bicarbonate, heavy metals and arsenic shall be conducted on each of the individual samples.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

B-5.1.1 *For Ammonium Bicarbonate*

From the test results for ammonium bicarbonate, the mean $\bar{X}$ and the range (R) of the test results shall be calculated (range being defined as the difference between the maximum and the minimum values of the test results). The lot shall be declared to have satisfied the requirement for this characteristic, if the value of the expression ($\bar{X} - 0.6\ R$) is greater than or equal to 98.0.

B-5.1.2 *For Heavy Metals and Arsenic*

The lot shall be declared as conforming to the requirements for these characteristics, if each of individual samples satisfies the test laid down in **A-7**.

B-5.2 For Composite Samples

The test results on the composite sample shall meet the corresponding requirements specified.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Food Additives Sectional Committee, FAD 08

Organization	*Representative(s)*
CSIR - Indian Institute of Toxicology Research, Lucknow	DR YOGESHWAR SHUKLA **(*Chairperson*)**
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